

This Page Is Inserted by IFW Operations  
and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representation of  
The original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning documents *will not* correct images,  
please do not report the images to the  
Image Problem Mailbox.**

**THIS PAGE BLANK (USPTO)**

# (12) UK Patent Application (19) GB (11) 2 270 030 (13) A

(43) Date of A Publication 02.03.1994

(21) Application No 9217599.1

(22) Date of Filing 19.08.1992

(71) Applicant(s)  
Courtaulds Fibres (Holdings) Limited

(Incorporated in the United Kingdom)

50 George Street, LONDON, W1A 2BB,  
United Kingdom

(72) Inventor(s)  
William Brunskill  
John Frederick Carefull  
Paul John Akers

(74) Agent and/or Address for Service  
J Y & G W Johnson  
Furnival House, 14-18 High Holborn, LONDON,  
WC1V 6DE, United Kingdom

(51) INT CL<sup>5</sup>  
D01D 10/02

(52) UK CL (Edition M )  
B5B BAH B350 B351 B363 B365 B367 B37Y B404 B901  
B902  
B2E EFA E1202 E1212 E1217 E1326 E489T

(56) Documents Cited  
None

(58) Field of Search  
UK CL (Edition K ) B5B BAH BCL BCM

## (54) Method of producing fibre or film

(57) A water-absorbent water-insoluble fibre or film is produced by extruding an aqueous solution of a water-soluble polymer into a gaseous medium to form fibre or film. The extruded fibre or film is collected at a moisture content of 8 to 25% based on the dry weight of the fibre or film and is further dried at a temperature no greater than 100°C before crosslinking the fibre or film at a temperature in the range 125 to 250°C to a degree sufficient that the crosslinked fibre or film is water-insoluble. The fibre or film entering the crosslinking step has a moisture content which is reduced, preferably by at least 5%, and which is in the range 0 to 10%. The heat-crosslinked fibre is preferably treated with moist air after crosslinking to raise the moisture content of the fibre to at least 10%, particularly if the fibre is for textile processing. Water-absorbent water-insoluble coatings can be produced by a similar process.

GB 2 270 030 A

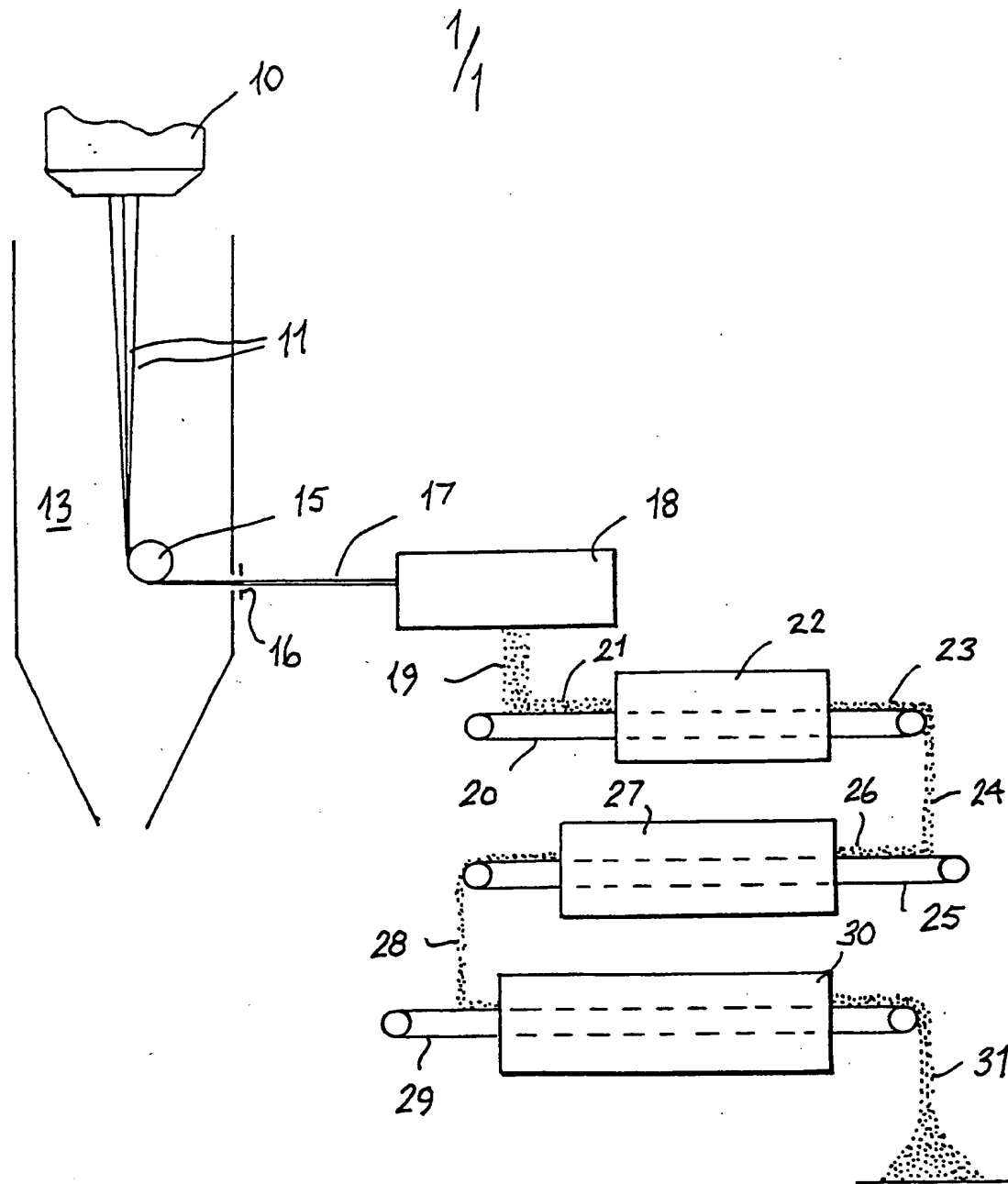


FIG. 1

Method of Producing Fibre or Film

This invention relates to methods of producing water-absorbent shaped articles such as fibre or film, including free-standing film and film coated on a surface.

5 It is known to produce fibre or film of polymeric material which is capable of absorbing large quantities of water or other liquids. One method of manufacturing such fibre or film is to use as a starting material a water-soluble polymer which can be crosslinked under the action of  
10 heat. The polymeric material is dissolved in water to form an aqueous solution which is then extruded into a heated gaseous environment. This produces fibre or film of the water-soluble polymer, which is dried by the heated gaseous environment into which the material is extruded. The  
15 extruded fibre or film is subsequently heated to crosslink the polymer to such a degree that it is water-insoluble but water-absorbent.

Without the crosslinking step the fibre or film will re-dissolve should it ever encounter water subsequently. The  
20 crosslinking step, therefore, stabilises the fibre or film so that on encountering water it absorbs the water without dissolving. The more the material is heated and crosslinked the less capable the product is of absorbing water, but in general terms the more stable the material is and the more  
25 it retains its structure on absorbing water, that is to say the more the fibre retains its fibrous nature. The exact amount of crosslinking and heating can be varied depending upon the uses to which the water-absorbent fibre or film is to be put.

30 EP-A-268498 describes a water-absorbent water-insoluble polymeric fibre, film, coating, bonding layer or foam, made by forming a substantially linear polymer by polymerisation of water-soluble ethylenically unsaturated monomer blends comprising carboxylic and hydroxylic monomers and then

reacting the carboxylic and hydroxylic monomers in the linear polymer to form internal crosslinks within the polymer.

EP-A-269393 describes a water-absorbent, water-  
5 insoluble crosslinked polymer fibre or film made by dry extrusion of a solution of a substantially linear polymer formed from a water-soluble blend of monoethylenically unsaturated monomers comprising a plasticising monomer, evaporating the solvent and forming polymeric fibre or film  
10 plasticised by an external plasticiser, stretching the fibre or film while the external plasticiser remains in the fibre or film, and then crosslinking the polymer.

EP-A-342919 describes film or fibre made by extrusion and stretching from a polymer of water-soluble ethylenically  
15 unsaturated monomers that include ionic monomer. A counterionic lubricant compound is absorbed into the surface of the fibre or film before or during the stretching.

EP-A-397410 describes a water-soluble linear polymer of carboxylic acid monomers such as acrylic acid and a  
20 hydroxylic monomer which can be crosslinked, after being shaped by extrusion of an aqueous solution of the polymer as fibre or film, to form crosslinks between the carboxyl and hydroxyl groups.

FR-A-2355929 describes a process for manufacturing  
25 yarns and fibres having a water-retention capacity of more than 80% by weight, in which aqueous solutions containing water-soluble polymers of acrylic acid, distributed homogeneously, and aliphatic, cycloaliphatic, araliphatic or aromatic compounds that contain at least two hydroxyl and/or  
30 primary or secondary amino groups in their molecule are spun, and the yarns spun in this way are heated at temperatures of between 100 and 200°C.

A method according to the invention of producing

water-absorbent water-insoluble fibre or film, by extruding an aqueous solution of a water-soluble polymer into a gaseous medium to form fibre or film and crosslinking the fibre or film at a temperature in the range 125-250°C to a degree sufficient that the crosslinked fibre or film is water-insoluble, is characterised in that the extruded fibre or film is collected at a moisture content of 8-25% based on the dry weight of the fibre or film and is further dried at a temperature no greater than 100°C so that the fibre or film entering the crosslinking step has a lower moisture content in the range 0-10% based on the dry weight of the fibre or film.

The conventional way of processing the fibre or film to crosslink the polymer would be to collect the fibre or film at a moisture content at which the fibre or film is no longer self-adherent, and then to heat the fibre or film at a temperature in the range 125 to 250°C to cause crosslinking. It has unexpectedly been found according to the invention that, if the fibre or film is dried at a temperature below 100°C after collection but before passing into the crosslinking zone, then a tougher material can be obtained, in the sense that it is less prone to damage during subsequent processing such as carding and crimping of fibre. This is surprising, since the fibre or film will in any event be dried as it is heated during the crosslinking stage.

Preferably, the non-crosslinked polymer is substantially linear and is formed from a water-soluble blend of monoethylenically unsaturated monomers that must be selected such that the final crosslinked polymer is water-absorbent. Ways of selecting monomers for this purpose are known, for example from EP-A-397410 mentioned above. Preferably, the water-soluble blend of monoethylenically unsaturated monomers includes an anionic monomer, for example it comprises 50 to 95% by weight ethylenically unsaturated carboxylic monomer and 5 to 50% by weight

copolymerisable ethylenically unsaturated monomer. The copolymerisable monomer preferably comprises mainly a non-ionic monomer. The monomers used in the invention are preferably acrylic monomers.

5 Preferred carboxylic monomers are methacrylic acid or acrylic acid, but maleic acid or anhydride, itaconic acid or any of the other conventional ethylenically unsaturated carboxylic acids or anhydrides are also suitable. Some of the carboxylic monomer units in the copolymer can optionally  
10 be replaced by monomer units derived from an ethylenically unsaturated sulphonic acid such as 2-acrylamido-2-methylpropane sulphonic acid or allyl sulphonic acid. Carboxylic and sulphonic monomers may be present in the final polymer in free acid or water-soluble salt form,  
15 suitable salts being formed with ammonia, an amine or an alkali metal.

When the crosslinking reaction involves reaction with the carboxylic acid groups it is usually preferred that at least some of the carboxylic acid groups should be present  
20 as free acid groups before the crosslinking occurs. For instance, for this purpose, it may be adequate for 10 to 75%, preferably 25 to 75%, of the acid groups to be in free acid form before the crosslinking occurs.

The copolymerisable ethylenically unsaturated monomer  
25 for copolymerisation with the carboxylic monomer may be a water-soluble ethylenically unsaturated monomer such as acrylamide or a water-insoluble monomer, for example an olefin, such as isobutylene, an aromatic ethylenically unsaturated monomer, such as styrene or a substituted  
30 styrene, an alkyl ester of acrylic or methacrylic acid, such as methyl or ethyl acrylate or methacrylate, butyl acrylate or methacrylate or 2-ethylhexyl acrylate or methacrylate, vinyl acetate or acrylonitrile. One or more copolymerisable monomers may be present. A monomer that will provide groups  
35 for internal crosslinking with the carboxylic groups (as



discussed below) is usually included. Other non-ionic monomers that may be used include ethylenically unsaturated monomers that carry a pendent group of the formula  $-A_m B_n A_p R$  where B is ethyleneoxy, n is a integer of at least 2, A is propyleneoxy or butyleneoxy, m and p are each an integer less than n and preferably below 2 and most preferably zero, and R is a hydrophobic group containing at least 8 carbon atoms as described in more detail in EP-A-213799. The comonomer(s) are generally present in amounts of at least 5% and preferably at least 10% by weight based on the monomers used for forming the copolymer, and they may be present in amounts up to about 50%, generally below 45%, by weight.

The substantially linear water-soluble polymer may be formed from the monomer blend in any conventional manner. It may be preformed and then dissolved to form a polymer solution. For instance, it may be made by reverse-phase polymerisation if the monomer blend is soluble in water or by water-in-oil emulsion polymerisation if the blend is insoluble in water, e.g. at a low pH. However, this can incur the risk that the polymer may be contaminated by surfactant, and this is undesirable. Preferably, therefore, the polymer is made by aqueous solution polymerisation or other solution polymerisation methods. Generally, it is formed by solution polymerisation in the solvent in which it is to be extruded (usually water). The polymerisation can be conducted in a conventional manner in the presence of conventional initiators and/or chain-transfer agents to give the desired molecular weight.

The concentration of polymer in the aqueous solution to be passed through the spinneret is generally in the range 5 to 50% by weight and will be selected, having regard to the molecular weight of the polymer, so as to give a solution having a viscosity that is convenient for extrusion. The spinneret can be of the type conventionally used in synthetic fibre production. The concentration of polymer is usually at least 15% by weight, with values of

30% to 45%, e.g. 35% to 40%, by weight often being particularly suitable.

The solution that is extruded may have a viscosity as low as, for instance, 20,000 mPa.s at 20°C but generally the viscosity is at least 50,000 and usually at least 80,000 and sometimes as high as 200,000 mPa.s, as measured at 20°C using a Brookfield RVT spindle 7 at 20 rpm. The viscosity desirably is also relatively high at the extrusion (spinning) temperature, which typically is elevated, for instance above 80°C but below the boiling point of the copolymer solution. The solution at 80°C usually has a viscosity of at least 5,000 or 10,000 mPa.s and most preferably at least 20,000 mPa.s. For instance it may be in the range 50,000 to 100,000 mPa.s. These values may be obtained by extrapolation from values obtained using a Brookfield RVT viscometer spindle 7 at 20 rpm at a range of temperatures below 80°C.

The solvent of the solution that is extruded is generally water but can be a blend of water and organic solvent, for example methanol. The solvent must be volatile so as to permit rapid evaporation after extrusion. The gaseous environment into which the solution is extruded is preferably hot air. When forming fibre, the hot air can be contained in a cell of the type conventionally used for dry spinning. The extruded fibre can be taken up on conventional textile machinery, such as a godet, as a yarn or tow. A conventional spin finish, which is preferably non-aqueous, is usually applied to the fibre before it is taken up.

The moisture content of the fibre at the point at which it is taken up is generally in the range 8 to 25% based on the dry weight of fibre. The fibre at this point preferably has a moisture content of at least 10%, most preferably at least 12%, by weight, particularly if the fibre has to be handled mechanically before it passes to the

drying step. If the fibre is dried to a moisture content less than 8% before collection, it has decreased mechanical strength and in particular is brittle. It may not withstand processes such as cutting, crimping or cross-laying to form  
5 a web. The moisture content of the fibre is preferably no higher than 22%, based on the dry weight of fibre, to avoid fibres sticking together if they are collected on a package. A moisture content of at least 15% and no more than 20% is particularly advantageous.

10        When forming film, the aqueous solution can for example be extruded via a slit die or an annular die through a heated gaseous environment, generally hot air, on to a support surface, for example a heated rotary drum. The support surface has release properties. Drying of the film  
15 continues on the drum, and the film is stripped from the drum and taken up on rolls. The moisture content of the film as it is taken up is generally in the range 8 to 25% based on the dry weight of film, most preferably 10 to 20%.

      It is preferred to stretch the fibre or film before it  
20 is collected. Stretching is effected by having the speed of the collection apparatus, for example the take-off godet, higher than the extrusion rate of the polymer solution (the linear velocity of the polymer solution through the exit capillary of the spinneret or die). The ratio of the take-  
25 off speed to the extrusion speed is generally up to 10:1 but is preferably in the range 2-8:1, most preferably 3-6:1.

      The diameter of the fibre as it is collected preferably corresponds to a weight of not more than 20  
decitex per filament, for example in the range 2 to 15  
30 decitex per filament.

      The fibre or film thus taken up is further dried to a moisture content in the range 0-10% based on the dry weight of fibre or film. The degree of drying carried out in this further drying step is preferably such as to reduce the

moisture content of the fibre or film by at least 5% based on the dry weight of fibre or film. Drying is carried out at a temperature no greater than 100°C to avoid damage to the fibre or film by the formation of bubbles of superheated steam within the fibre or film structure. Drying is preferably carried out by passing the fibre or film through an oven, generally at a temperature of above 50°C, for example at a temperature in the range 60 to 90°C.

If any mechanical process step needs to be carried out on the fibre or film, it may be advantageous to do it before the further drying step while the fibre or film has a moisture content of at least 8%, preferably at least 12%, to 25%, based on the dry weight of fibre or film, most preferably 15-20%. Examples of such mechanical process steps are crimping of fibre, stretching of fibre or film, cutting of fibre into staple fibre, pneumatic conveying, fibre blending, crosslaying of fibre to form a batt or slitting of film longitudinally to form tapes. For example, the fibre can be cut into staple fibre and dried as a bed of staple fibre on a foraminous support, preferably a moving belt; alternatively continuous fibre can be cross-laid on a foraminous belt to form a batt and dried in that form. Alternatively, mechanical treatment can be carried out during the further drying step. For example a film can be dried while being stretched on a stenter.

In the crosslinking step, the water-soluble polymer is heated to cause crosslinking of the polymer chains sufficient to render the polymer water-insoluble. The crosslinking can be effected by reaction into the backbone of the water-soluble polymer but preferably is effected by crosslinking through pendent groups provided by one or more of the monomers that have been polymerised to form the water-soluble polymer. The crosslinking preferably comprises the formation of ester, amide (or imide) or urethane groups by reaction of pendent carboxylic acid groups in the water-soluble polymer after extruding the

copolymer. Formation of ester groups is preferred.

Preferably, the polymer is internally crosslinked by reaction between reactive groups within the extruded copolymer. Usually, the carboxylic groups act as one type of reactive group and are reacted with hydroxyl, epoxide, amino or blocked isocyanate groups. Particularly preferred systems are described in detail in EP-A-268498. In these systems the extruded copolymer is formed from a monomer blend comprising monomer that provides carboxylic acid monomer groups and monomer that provides hydroxyl groups that can react with the carboxylic acid groups to form ester crosslinkages that contain only carbon and oxygen atoms in the linkages, and these carboxylic and hydroxyl groups are reacted after extrusion to form the said crosslinkages. Generally, the carboxylic acid groups are provided by acrylic acid or methacrylic acid and the hydroxyl groups are provided by allyl alcohol, an epoxide-substituted vinyl monomer such as glycidyl methacrylate or a hydroxyalkyl ester of a vinyl carboxylic acid such as 2-hydroxyethyl acrylate, 2-hydroxyethyl methacrylate, 2-hydroxypropyl methacrylate or 3-hydroxypropyl methacrylate, or by vinyl alcohol groups. Alternative hydroxyl group-containing monomers are those of the formula  $\text{CHR}^1=\text{CR}^2-\text{Y}-\text{M}_a-\text{OH}$ , where  $\text{R}^1$  is hydrogen or carboxyl,  $\text{R}^2$  is hydrogen or methyl, Y is O,  $\text{CH}_2\text{O}$  or  $\text{COO}$ , M is alkyleneoxy, for example ethyleneoxy or 1,2-propyleneoxy, and a is an integer greater than 1 and preferably at least 5, as disclosed in EP-A-397410. Alternatively, the comonomer can contain a primary or secondary amino group, for example 2-aminoethyl methacrylate, which reacts to form an amide crosslink, or it can contain an isocyanate group (which may need to be blocked to prevent crosslinking during extrusion), for example 2-isocyanatoethyl methacrylate, to form urethane crosslinks.

Alternatively, reaction may be with an external crosslinking agent which should be substantially non-

volatile at the temperatures reached in the extrusion process. Various systems for externally crosslinking the copolymer are described in EP-A-269393 and FR-A-2355929 and these can be used in the present invention. For example, a 5 hydroxy-functional or carboxyl-functional linear polymer can be crosslinked by a diisocyanate (which may need to be blocked) or a carboxy-functional polymer can be crosslinked by a polyamine such as ethylene diamine or by a polyfunctional reagent containing hydroxyl and/or epoxide 10 groups.

The temperature used to crosslink the fibre or film is generally in the range 125 to 250°C. When forming ester crosslinks by the reaction of carboxylic acid and hydroxyl groups, the preferred temperature of crosslinking is at 15 least 150°C or 160°C, and is preferably no higher than 225°C. The temperatures reached during the extrusion process, for example the temperature reached by filaments during dry spinning, are lower than the crosslinking temperature, preferably at least 30°C lower, to prevent 20 premature crosslinking; when forming ester crosslinks, the temperature reached during the extrusion process is preferably no higher than 120°C. The time for which the crosslinking step is carried out is generally in the range 2 minutes to 2 hours; crosslinking times of 5 to 15 minutes 25 are preferred unless the temperatures required to effect crosslinking in this time might damage any chemical materials present in the fibre or film.

The moisture content of the fibre or film as it enters the heat zone used for crosslinking is in the range 0-10% 30 based on the weight of fibre or film. We have found that, if the moisture content of the fibre or film is above 10% at this stage, damage to the structure of the fibre or film is caused by the rapid vapourisation of moisture at the high temperatures used for crosslinking. In general, the 35 strength of the fibre produced increases with a decreasing moisture content of the fibre or film on entering

crosslinking. Decreasing moisture content leads to fibre or film of improved mechanical properties down to a moisture content of 4 or 5%. Thus in the most preferred method of carrying out the invention, fibre is dry spun and collected at a moisture content of 15 to 20% based on the dry weight of fibre, is crimped and/or cut as required, and is dried to a moisture content of 4 to 8% before entering the crosslinking step. However, if the fibre is initially collected at a moisture content of 10-12%, it is still advantageous to dry the fibre to a moisture content of 3-6% before entering crosslinking; similarly it is advantageous to dry from a moisture content of 15-20% down to 10% before crosslinking, although drying to a lower level will give further improvement in fibre quality. In general, there is little extra advantage in drying to a moisture content of below 4%, and particularly below 2%, compared to the extra cost of removing these low levels of moisture in a drying step carried out at below 100°C.

The fibre or film emerging from the crosslinking step has a very low moisture content, generally less than 1%, because of the high temperatures used in crosslinking. For some uses, particularly fibre which is to undergo further textile processing, it may be preferred to add moisture in a controlled manner after crosslinking, for example to increase the moisture content to a value of at least 10%, for example 10 to 20%, based on the dry weight of fibre, to give a less brittle fibre.

According to another aspect of the invention, a similar process is used to produce a water-absorbent water-insoluble coating. A flexible substrate such as a film or fabric is coated with an aqueous solution of a crosslinkable water-soluble polymer. The coating is dried, generally at a temperature of above 100°C, to a moisture content of 8-25% by weight, then further dried at a temperature no greater than 100°C to a lower moisture content in the range 0-10% by weight. The further dried coating is crosslinked by heating

at a temperature in the range <sup>125</sup>~~100~~-250°C. The coated film or fabric can be mechanically treated, for example wound on a package, stretched and/or cut, between the two drying steps. The crosslinkable water-soluble polymer used can be as  
5 described above for use in preparing fibre or film, and it can be coated on the flexible substrate as an aqueous solution of similar concentration and viscosity.

The invention will now be described by way of example with reference to the single figure of the accompanying  
10 drawing, which is a schematic view of a process for dry spinning, collecting, cutting, drying, crosslinking and remoisturising fibre according to the invention.

A heated and filtered spinning dope is extruded through spinneret 10 to form fibre 11 in a dry spinning cell  
15 13. Hot air circulates in the cell 13. The extruded fibre 11 is dried and solidified as it passes down the cell 13 and is collected around a godet 15. The fibre emerges from the side of the drying chamber of the cell 13, through a small hole 16, in the form of a tow 17.

20 The moisture content of the fibre emerging at 16 in the form of a tow is 8 to 25 per cent based on the dry weight of fibre. If required, a spin finish can be applied to the fibre strands as they are emerging in the form of tow 17.

25 The tow 17 passes into a conventional staple cutting machine 18 and falls from the cutter as staple fibre 19. The staple fibre 19 falls onto a moving belt 20 and forms a bed 21 of fibre having a moisture content in the range 8 to 25 per cent. The fibre on the bed is then passed through a  
30 drying oven 22 where it is dried at a temperature of no more than 100°C, for example 80°C, for a time sufficient to give a moisture content of 0 to 10 per cent, for example about 5 per cent. The dried fibre emerges at 23 and falls from the belt 20 in a stream 24 onto a second conveyor 25. The



predried staple fibre forms a bed 26 on the conveyor 25 and the bed 26 is then passed through an oven 27 where the predried staple fibre is heated at a temperature in the range 125 to 250°C, for example at 200°C for 8 minutes, to crosslink the water-soluble polymer to form the absorbent fibre. After crosslinking, the absorbent fibre is fed off the conveyor 25 as at 28 onto a further conveyor 29.

On the conveyor 29 the bed of fibrous absorbing material is passed through a remoisturising chamber 30 where the moisture content of the fibre is increased to at least 10 per cent, for example approximately 15 per cent. The remoisturised fibre is then passed off the conveyor 29 as at 31 to suitable storing and/or packaging equipment.

The water-absorbent water-insoluble fibre or film of the present invention can be used in various products. The fibre can, for example, be used in absorbent personal products such as tampons, disposable diapers, sanitary napkins or incontinence pads. The absorbent fibre is preferably used in combination with other fibres, for example cellulosic fibre such as cotton or regenerated cellulose fibre, including multi-limbed cellulose fibre as described in EP-A-301874, or polypropylene or polyester fibre. The absorbent fibre can be intimately mixed with said other fibre, for example by carding or air-laying the fibres together to form a web of mixed fibres. Alternatively, the absorbent fibre can be used as a layer, for example a non-woven fabric, of absorbent sandwiched between layers of other fibre. An absorbent film produced according to the invention can similarly be used as an absorbent sandwiched between fibrous layers. The proportion of absorbent fibre in a blend with cellulosic fibre for absorbent products can for example be at least 5% and up to 95% by weight, preferably at least 10% and up to 50% by weight. The absorbent fibre can also be used at similar levels in conjunction with fluffed wood pulp or synthetic fibre pulp, for example polyolefin pulp, in absorbent products.

A yarn, woven fabric or nonwoven fabric comprising the absorbent fibre, or an absorbent film produced according to the invention, can be used as a swellable material which prevents ingress of water in underground cables. A yarn or  
5 fabric tape can be used to wrap cable or can be laid longitudinally in the cable.

The absorbent fibre or film can be used in many other applications of the types described in Research Disclosure, January 1992 at pages 60-61, for example in filters,  
10 absorbent liners or mats for packaging, disposable wipes, mats, shoe insoles or bed sheets, swellable gaskets or seals, moisture-retention mats in horticulture, moisture-retaining packaging or swellable self-sealing stitching threads.

15 The invention is illustrated by the following Examples, in which parts and percentages are by weight unless otherwise stated:-

#### Example 1

A 38% aqueous solution of a copolymer of 78 mole %  
20 acrylic acid (75% neutralised as sodium salt), 20 mole % methyl acrylate and 2 mole % hexapropylene glycol monomethacrylate was spun into fibre through a spinneret into a cell where water was evaporated from the fibre. The temperature of the solution at the spinneret was between 90  
25 and 100°C. The cell was heated by tube wall heaters at 150°C. The fibre was collected as a tow at approximately 200 m/min and was passed to a staple cutter. The fibre had a moisture content of 16% based on dry weight of fibre as it passed to the staple cutter and a weight per filament of  
30 approximately 15 dtex.

The fibre was cut to a length of 160 mm and was dried in a forced air oven at 70°C to a moisture content of 7% based on dry weight of fibre. The dried fibre was then

crosslinked in an oven at 200°C for 10 minutes to form a water-absorbent water-insoluble fibre. The fibre was then conditioned at 60°C and 50% relative humidity for 8 hours to a moisture content of 15%.

- 5        The fibre produced had a free swell absorbency of 50 g/g with a retention under load of 35 g/g.

10        In the free swell absorbency test. 0.5g fibre is dispersed in 30 ml 0.9% by weight aqueous saline solution and left for 5 minutes. The dispersion is then filtered through a sintered Mark 1 funnel of pore size 100-160 microns and is left for 5 minutes or until it stops dripping. The amount of water filtered through the funnel is weighed and the weight of water absorbed by the fibres is calculated by subtraction.

- 15        In addition to the above test, the retention of the saline solution after application of pressure at about 3.4 kPa for 5 minutes or until dripping stops is measured by weighing the water expressed after application of pressure.

20        For comparison a control fibre was produced in the same way except that the drying step was omitted. Each fibre sample was blended in equal proportions with 10 dtex 153 mm polyester staple fibre and processed through semi-worsted carding and gilling equipment to produce a sliver with a weight of 11.5 g/m. The amount of short, broken  
25 fibres deposited under the card and on the surfaces after the exit nips from the card and the gilling equipment was significantly less compared to the control.

CLAIMS

1. A method of producing water-absorbent water-insoluble fibre or film by extruding an aqueous solution of a water-soluble polymer into a gaseous medium to form fibre or film  
5 and crosslinking the fibre or film at a temperature in the range 125-250°C to a degree sufficient that the crosslinked fibre or film is water-insoluble, characterised in that the extruded fibre or film is collected at a moisture content of 8-25% based on the dry weight of the fibre or film and is  
10 further dried at a temperature no greater than 100°C so that the fibre or film entering the crosslinking step has a lower moisture content in the range 0-10% based on the dry weight of the fibre or film.

2. A method according to claim 1 of producing water-  
15 absorbent water-insoluble fibre, characterised in that the aqueous solution of water-soluble polymer is extruded through a spinneret to form fibre and the fibre is collected as a tow or yarn at a moisture content of 10-22% based on the dry weight of fibre.

20 3. A method according to claim 2, characterised in that the fibre has a decitex per filament of 2 to 20.

4. A method according to claim 2 or claim 3, characterised in that the tow or yarn is cut into staple fibre at a moisture content of 10-22% based on dry weight of  
25 fibre and is subsequently dried and crosslinked in fibrous form.

5. A method according to claim 4, characterised in that the staple fibre is dried as a bed of fibre on a moving belt.

30 6. A method according to any of claims 2 to 5, characterised in that the fibre is re-moisturised after

crosslinking to a moisture content of at least 10% based on the dry weight of fibre.

7. A method according to claim 1 for the production of water-absorbent water-insoluble film, characterised in that  
5 the aqueous solution of water-soluble polymer is extruded through a slit die or annular die to form a film which is collected at a moisture content of 10-22% based on dry weight of film.

8. A method according to claim 7, characterised in that  
10 the film is longitudinally slit at a moisture content of 10-22% based on dry weight of film before the step of drying at a temperature no greater than 100°C.

9. A method according to any of claims 1 to 8,  
characterised in that the fibre or film is stretched before  
15 it is collected.

10. A method according to any of claims 1 to 9,  
characterised in that the extruded fibre or film is collected at a moisture content of at least 12% based on dry weight of fibre or film.

20 11. A method according to claim 10, characterised in that the extruded fibre or film is collected at a moisture content of 15-20% based on dry weight of fibre or film.

12. A method according to any of claims 1 to 11,  
characterised in that the extruded fibre or film is dried to  
25 a moisture content of 4-8% based on dry weight of fibre or film at a temperature no greater than 100°C before entering the crosslinking step.

13. A method according to any of claims 1 to 12,  
characterised in that the moisture content of the fibre or  
30 film is reduced by at least 5% based on the dry weight of fibre or film during the step of further drying at a

temperature no greater than 100°C.

14. A method according to any of claims 1 to 13, characterised in that the said drying step is carried out at a temperature of 60-90°C.

5 15. A method according to any of claims 1 to 14, characterised in that the aqueous solution has a concentration of water-soluble polymer of 30-45% by weight and is extruded at a temperature which is above 80°C but below the boiling point of the copolymer solution.

10 16. A method according to any of claims 1 to 15, characterised in that the water-soluble polymer is a copolymer of 50-95% by weight ethylenically unsaturated carboxylic monomer and 5-50% by weight copolymerisable enthylenically unsaturated monomer.

15 17. A method according to claim 16, characterised in that the copolymerisable ethylenically unsaturated monomer consists at least partly of a hydroxy-functional or epoxide-functional comonomer so that ester crosslinks are formed by reaction between carboxylic acid groups derived from the  
20 carboxylic monomer and hydroxyl or epoxide groups derived from the comonomer during the crosslinking step.

18. A method of producing a water-absorbent water-insoluble coating, characterised in that a flexible substrate is coated with an aqueous solution of a  
25 crosslinkable water-soluble polymer and the coating is dried to a moisture content of 8-25% by weight, then further dried at a temperature no greater than 100°C to a lower moisture content in the range 0-10% by weight and the further dried coating is crosslinked by heating at a temperature in the  
30 range <sup>125</sup>~~100~~-250°C.

**Patents Act 1977**  
**Examiner's report to the Comptroller under**  
**Section 17 (The Search Report)**

-19-

Application number

GB 9217599.1

**Relevant Technical fields**

(i) UK Cl (Edition K ) B5B (BAH BCL BCM)

(ii) Int Cl (Edition 5 ) D01D

**Databases (see over)**

(i) UK Patent Office

(ii)

Search Examiner

DR J RIDDOCH

Date of Search

30 NOVEMBER 1992

Documents considered relevant following a search in respect of claims ALL

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
	NONE	

Category	Identity of document and relevant passages -20-	Relevant to claim(s)

#### Categories of documents

**X:** Document indicating lack of novelty or of inventive step.

**Y:** Document indicating lack of inventive step if combined with one or more other documents of the same category.

**A:** Document indicating technological background and/or state of the art.

**P:** Document published on or after the declared priority date but before the filing date of the present application.

**E:** Patent document published on or after, but with priority date earlier than, the filing date of the present application.

**&:** Member of the same patent family, corresponding document.

**Databases:** The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).